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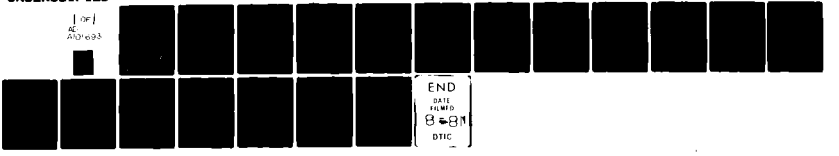

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INVESTIGATION OF QUANTUM EFFECTS IN HETEROSTRUCTURES

FINAL REPORT,

Leo Esaki

March 31, 1981

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Semiconductor superlattices made of periodic, ultra-thin layers of $In_{1-x}Ga_xAs$ and $GaSb_{1-y}As_y$ are grown by molecular beam epitaxy. The epitaxial process is established to provide atomically smooth interfaces and precisely controlled thickness and composition. Quantum states or subbands are created, which have been investigated experimentally and analyzed theoretically. Unique properties of this superlattice system, such as the semiconductor-semimetal transition and the spatial separation of carriers, have been

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observed. Energy positions and dispersion of the subbands and the existence of multiple subbands have been studied through a variety of physical measurements, primarily, magneto-oscillations and absorptions. The effect of the superlattice potential on the band structure at different points in the momentum space has also been demonstrated from electroreflectance measurements for both the $In_{1-x}Ga_xAs$-$GaSb_{1-y}As_y$ and the GaAs-$Ga_{1-x}Al_xAs$ system.

I. Statement of Objective

The project was undertaken to pursue studies of heterostructures and superlattices of semiconductors. The characteristic dimension of these structures is smaller than the mean free path of electrons so that the entire electron system is in the quantum regime. Quantum states or subbands for both electrons and holes are created, and they are dominant in governing the electronic properties. The focus of the investigation is on the specific superlattice system made of InAs and GaSb, which, because of the rather unique bandedge alignment at the interface of these two semiconductors, offers unusual characteristics in terms of subband energies and carrier transport.

II. Summary of Achievement

The recognition that the conduction bandedge of InAs is located in energy very close to and in fact below the valence bandedge of GaSb provided the original motivation for the investigation of heterostructures of these materials. The close vicinity of these bandedges results in their strong interaction, which is expected to play a major role in determining the subband configuration. The relative position of these bandedges is expected to give rise to a potential profile which not only confines electrons and holes in spatially separated regions but also creates a situation whereby the superlattice behavior changes from being a semiconductor to being a semimetal as the layer thickness is increased. All these features have been analyzed theoretically and observed experimentally.

Our project started with the molecular beam epitaxy of InAs and GaSb, and, indeed, of their alloys with GaAs, i.e. $In_{1-x}Ga_xAs$ and $GaSb_{1-y}As_y$. The lattice constants of the pure compounds are closely matched and can be so maintained in the alloys by controlling the compositions, x and y. After the growth conditions were established to yield high-quality films and heterostructures, single heterojunctions were fabricated to verify the bandedge relationship, and double-barrier structures were made to demonstrate the formation of quantum states. Subsequent measurements of optical absorptions and magneto-oscillations were performed on superlattices to investigate the subbands and their associated two-dimensional electron system. A large number of superlattices with different layer thicknesses were prepared and studied. From both transport and optical measurements under magnetic fields, the semimetallic properties were extensively explored, including the direct observation of the semiconductor-semimetal transition, the semimetallic behavior in the heterojunction limit, and detailed information about the subbands and their energy dispersion as well as the existence of multiple subbands. These are the major points to be described in the following. The knowledge we gained through this project far exceeds our original expectation.

(A) Theoretical Consideration

From a theoretical point of view, the present superlattice can be distinguished from that of GaAs-$Ga_{1-x}Al_xAs$ by the relative locations of the bandedge energies in the two host semiconductors. In GaAs-$Ga_{1-x}Al_xAs$, the conduction and valence bands are widely separated so that the subband

energies and their dispersion relation can be obtained by wavefunction matching along the direction perpendicular to the superlattice layers, using simple plane waves and treating electrons and holes separately. This procedure is no longer valid in InAs-GaSb or $In_{1-x}Ga_xAs$-$GaSb_{1-y}As_y$ because of the strong band interaction between the host materials. The situation is analyzed instead by use of Bloch functions in the k•p framework, assuming coupling between electrons and light holes. The results are quite general in that they reduce to those obtained previously for GaAs-$Ga_{1-x}Al_xAs$ as band interaction becomes negligible. Alternatively, the subband configuration can also be solved by the tight-binding method of linear combination of atomic orbitals. It gives essentially the same result as that from the wavefunction matching.

While the present superlattice system shows, expectedly, some similarities to the GaAs-$Ga_{1-x}Al_xAs$ system in terms of variations of subband energies and widths with layer thickness, the energy dispersion is usually more nonlinear and the effect of nonparabolicity in the conduction band of InAs is much larger. More significantly, there exists an energy range in superlattices of InAs-GaSb and $In_{1-x}Ga_xAs$-$GaSb_{1-y}As_y$ of low alloy compositions where both electron and hole states can be present simultaneously. As the layer thickness is increased, the superlattice energy gap decreases in magnitude and may become zero and eventually negative, in contrast to the situation in GaAs-$Ga_{1-x}Al_xAs$ whose energy gap is limited to that of GaAs. This results in a semiconductor-to-semimetal transition. The critical layer thickness of InAs at which the transition occurs is calculated to be 85Å

In the semimetallic regime, carrier transfer takes place: electrons in the valence band of GaSb flow to the conduction band of InAs and leave behind an equal number of holes. This transfer of carrier causes band bending, which in turn affects the subband energies. The situation is analyzed in a self-consistent fashion by use of the Thomas-Fermi approximation, taking into account the two-dimensional density of states. As the layer thickness is further increased, each interface becomes essentially independent and the superlattice reaches the heterojunction limit. Throughout the semimetallic regime, the carrier mobility is rather high, since it is introduced by the transfer process, not suffering from impurity scattering.

The other important difference between the InAs-GaSb superlattice and the GaAs-$Ga_{1-x}Al_xAs$ superlattice is the spatial separation of electrons and holes in the former system. Their associated wavefunctions are mismatched in space. Inter-subband transitions can be observed only when the wavefunctions of electrons and holes penetrate into adjacent regions where they overlap. Numerical calculations have shown that, in the present type of superlattices, the absorptions are weak in comparison with those of GaAs-$Ga_{1-x}Al_xAs$ and that contributions from subbands of the same or different energy indices are comparable. The superlattice energy gap invariably corresponds to the energy difference between the ground subbands of electrons and heavy holes. Transitions at high energies are possible for both light and heavy holes with the latter usually playing a dominant role.

(B) Growth and Evaluation

The MBE system used for the growth of GaAs and $Ga_{1-x}Al_xAs$ was modified by the addition of sources of In and Sb. The shutter operation was controlled either by a computer or a simple timer as the fluxes effused from the source ovens are remarkably stable. Growth rates are determined by the Group-III elements which have a sticking coefficient of unity, while the Group-V elements supplied are at least twice in flux to ensure stoichiometry. The compositional control in $In_{1-x}Ga_xAs$ can be readily achieved by adjusting the relative rates of In and Ga. For $GaSb_{1-y}As_y$, the situation is somewhat complex, as Sb plays a dominant role over As in competing for the adsorbed Ga; the controlling can best be achieved by maintaining the ratio of Sb/Ga flux below unity.

The deposition was typically done on (100) GaSb substrates at a temperature of ~450°C and a growth rate of ~2Å/sec. Substrates of (100) GaAs can also be used. The lattice mismatch of InAs and GaSb with respect to GaAs in this case requires a buffer growth of compositional grading to ensure high-quality films. We have found that a step-grading technique, for example, the use of successive layers of $In_{1-x}Ga_xAs$ with x=1, 0.75, 0.5, 0.25, and 0, is very effective in reducing the misfit dislocations and enhancing the electron mobilities of InAs films grown on GaAs. Typical background concentrations, arising from unknown, residual impurities, are in the region of $10^{16}/cm^3$, electrons for InAs and holes for GaSb. The electron mobility varies in the range of $10^3 cm^2/V\text{-sec}$ to beyond $10^4 cm^2/V\text{-sec}$. The impurity,

Sn, is usually used for doping purpose, which behaves as a donor in $In_{1-x}Ga_xAs$ throughout the entire alloy range, but as an acceptor in $GaSb_{1-y}As_y$ for $y < 0.2$ and a donor otherwise.

The smoothness of the overgrowth layers, the most important criterion for the success of the superlattice, is monitored during deposition by the high energy electron diffractometer (HEED). Elongated reconstruction patterns were observed indicating a microscopically smooth surface. Under the usual growth conditions of Group-V stabilized surface, the reconstructed pattern is c(2x8) for $In_{1-x}Ga_xAs$, similar to that for GaAs, but becomes c(2x6) for $GaSb_{1-y}As_y$ for $y \leq 0.2$. What is more important is the observation of the pattern changes at the onset of heteroepitaxy: One characteristic elongated pattern is changed instantly to another, if the heteroepitaxial layers have a lattice mismatch smaller than ~2%. This change, without an intervening, spotty pattern representative of a rough nucleation process, indicates an abrupt interface through which the smoothness is maintained.

External evaluations after the depositions are completed and the sample removed from the MBE system were performed by use of x-ray diffraction, optical absorption and nuclear backscattering. For uniform films of both $In_{1-x}Ga_xAs$ and $GaSb_{1-y}As_y$, the lattice constants and the energy gaps are determined throughout the entire alloy ranges. For the InAs-GaSb superlattice, the periodicity is clearly revealed from backscattering spectra using He ions. Additional channeling experiments have shown a high dechanneling rate along <110> than along <100> directions. These experiments, plus other

observations with regard to beam energy dependence and planar vs. axial dechanneling measurements, are explained on the basis of a lattice relaxation effect at the interface. No dangling bonds or defects are apparently present.

(C) Electronic Properties

The single most important parameter, on which our investigation of the InAs-GaSb system is hinged, is the energy difference of the valence bandedge of GaSb above the conduction bandedge of InAs. This parameter was neither quantitatively known nor qualitatively established at the start of our project; theoretical calculations vary in their predictions as to the relative positions of these two bandedges. Aimed at clarifying this point, we started with measuring the current-voltage characteristics of simple heterojunctions of n-$In_{1-x}Ga_xAs$-p-$GaSb_{1-y}As_y$ with x,y varying between 0 and 0.6. In the latter case, rectifying behavior was observed, indicating the formation of depletion regions at the interface as in ordinary p-n junctions. The rectification decreased with decreasing x and y, and an ohmic behavior was observed as the compositions approached those corresponding to InAs-GaSb. These unusual observations can only occur if carriers are accumulated at both sides of the interface, an indication, in a qualitative fashion, that the InAs conduction bandedge is below the GaSb valence bandedge.

Transport properties were subsequently investigated in double barriers for the purpose of observing energy quantization, a prerequisite for the formation of a superlattice. The structure consists of an $In_{1-x}Ga_xAs$ layer, serving as the potential well, sandwiched between two $GaSb_{1-y}As_y$ layers, as

barriers, and two outside $In_{1-x}Ga_xAs$ layers, doped to high electron concentrtions, as electrodes. Quantum or quasi-stationary states are formed in the well. The resonant condition of tunneling is fulfilled when the energy of the incident electrons from the electrode under applied voltages coincides with that of the quantum state. Using $x = y = 0.55$ and a well thickness from 40 to 70Å, prominent features were observed in the tunneling characteristics which can be identified with the quantum states. The results demonstrate the degree of perfection of the interface required for the pursuance of quantum effects in the superlattice.

With the experience gained through earlier investigations in the GaAs-$Ga_{1-x}Al_xAs$ superlattice, we proceeded systematically with experiments of optical absorption and magneto-resistance on the present system. Using a photon energy covering the range of 0.2 - 1.0eV, absorption coefficients were obtained in both compound and alloy samples with a layer thickness between 20 and 50Å. The absorptions are weak in all cases, consistent with theoretical considerations. The absorption edges are deduced, which, by comparing with theoretically calculated energy gaps of the superlattices, not only confirmed the validity of our theoretical model but also provided a quantitative determination of the relative bandedge energies. The crucial parameter, as mentioned above, was found to be 150 meV with the valence bandedge of GaSb above the conduction bandedge of InAs. More recent measurements on improved superlattices have, in addition, shown fine features in the absorption spectra at energies higher than the edges. These features in all cases can be identified

with high-energy subbands above the ground states. Contributions from both heavy and light hole subbands were observed. The overlap of their wavefunctions with that of the electrons has been shown to govern the intensities of the transitions.

Measurements of transverse magneto-resistance have been used extensively in the investigation of low-dimensional electron systems, for they probe directly into the anisotropic properties associated with the Fermi surface. Heavily doped semiconducting superlattices were initially used for this purpose. For a magnetic field up to 18T, pronounced Shubnikov-de Haas oscillations were observed. The subband energies and their associated electron densities are obtained from the field positions at which the oscillatory extrema occur. The essentially two-dimensional nature of the electrons is demonstrated directly from the orientation dependence of the field. Results in agreement with theoretical predictions have been obtained from a large number of samples, including situations where more than one subband are populated. Related experiments have led to other interesting observations: the observation of negative magneto-resistance, the determination of electron effective mass from the temperature dependence of the oscillations, and the observation of cyclotron resonance. The latter two measurements show the mass enhancement due to nonparabolicity effect of the conduction band of InAs.

The possibility of the semiconductor-semimetal transition has greatly attracted our attention, as it represents a remarkable situation where a semimetal can be achieved with two host semiconductors. The clear demonstra-

tion of such a transition was provided by Hall measurements which measure the enhanced electron concentrations from carrier transfer, as described in a previous section. For undoped superlattices with different layer thicknesses, the electron concentration was found to increase dramatically at a layer thickness of ~100Å, close to the onset of the transition predicted theoretically. Subsequent experiments of Shubnikov-de Haas oscillations gave a quantitative measure of the electron density or Fermi energy throughout the semimetallic region from the semiconducting limit with a layer thickness <100Å to the heterojunction limit with a layer thickness >1000Å The Fermi energy first increases with the layer thickness as it is dominated by the carrier transfer. The effect of band bending gradually becomes important, which pushes the electron subband upward in energy and the hole subband downward, resulting in a decrease in the Fermi energy with a further increase in the layer thickness. Eventually, the superlattice becomes in essence a series of heterojunctions, and both the subband energies and the Fermi level are saturated. The observed variation with thickness is in very good agreement with the calculated result.

The high electron mobility achieved in semimetallic superlattices have made it possible to observe well-defined oscillations at rather low magnetic fields below 1T. This enabled us to examine fine features in the oscillatory spectra, which arise from multiple subbands as well as subband dispersion. One of the interesting observations is another transition from semimetal to semiconductor by the application of magnetic fields. The fields raise the

Landau levels of electrons and lower those of holes. In a semimetallic superlattice, the ground Landau level of holes eventually becomes lower than the ground Landau level of electrons. The semimetallic carriers are depleted, and the superlattice, again, behaves as a semiconductor. This magnetic field-induced transition was observed in semimetallic samples with relatively thin layers, so that the condition of transition can be realized with available, moderate fields.

Recently, experiments of magneto-absorptions in the far infrared region were carried out, which provided a wealth of information about the semimetallic superlattice. Oscillatory absorption spectra were observed. They arise from inter-subband transitions from Landau levels of holes to those of electrons. The energy gap of the superlattice obtained is negative, demonstrating directly the semimetallic nature. Additional oscillations were also observed, and can be identified as due to the finite subband width and other subbands with high energies, depending on the configurations of the superlattice. These results and the results of cyclotron resonance, which were also measured in the semimetallic superlattices, can be fitted with theoretical calculations to give an overall picture of the subbands in the superlattice.

Our investigations of the quantum effect in superlattices were not limited to the Brillouin zone center. By observing transitions from electroreflectance measurements, we have for the first time carried the studies of the superlattice effect on the subband strcture at other points in the Brillouin zone. Both the $InGa_{1-x}As_x$-$GaSb_{1-y}As_y$ and the GaAs-$Ga_{1-x}Al_xAs$ superlat-

tices were investigated. The observed transition energies in the latter system are shifted to higher values with respect to those of bulk GaAs. These values agree very well with those calculated theoretically by use of a similar method to that at the zone center, with the exception of taking into account the different bandedge energies and appropriate effective masses. For the $In_{1-x}Ga_xAs$-$GaSb_{1-y}As_y$ system, however, corresponding shifts are negligible for x, y ≤ 0.2 but are very large for higher alloy compositions. These observations cannot be explained by the calculations used so successfully at the zone center, and call for more fundamental theoretical considerations beyond the one-dimensional model. It should be mentioned in this connection that transitions associated with the spin-split valence band were also observed by this electroreflectance technique. In fact, the same technique was also applied to Ge-GaAs superlattices, which we have successfully fabricated recently, as evaluated from nuclear backscattering experiments.

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